Present and Future Applications of 3DXRD within Materials Science

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1. Introduction

When developing the 3 Dimensional X-ray Diffraction (3DXRD) microscope, the aims were to achieve:

i) non-destructive bulk measurements (3D)

ii) spatial resolution matching typical micro-structural scales, i.e. μm scale

iii) measurements fast enough to follow typical dynamical processes in-situ, i.e. second-minutes time resolutions

This was to allow in-situ mapping of bulk microstructures in otherwise opaque materials.

2. The 3DXRD microscope

The 3DXRD microscope has since 2000 been in regular operation in the second hutch at the Materials Science Beamline ID11 at the European Synchrotron Radiation Facility (ESRF). This is a high energy beamline and the 3DXRD typically operates at 40-100 keV. This means penetration depths in the mm-cm range in typical materials. A sketch of the 3DXRD is shown in Fig.1. The incident monochromatic beam penetrates the sample and diffraction occurs from all microstructural elements within the gauge volume fulfilling the Bragg condition. The diffracted signals are recorded on a 2D detector. The complete microstructure and the crystallographic orientations of all its elements can be determined simply performing ω-scans (See Fig. 1). [eg. 1, 2].

Various methods exist to determine the third dimension, i.e. where along the x-direction (see Fig. 1) a diffracting element of the microstructure is positioned. The most straightforward is to use a slit after the sample, whereby the diffracting volume is defined at the cross beam position. A microstructure map is thus obtained by scanning the sample relative to this position. This method is, however, in general too slow for in-situ experiments. Instead a tracking method, recording the diffraction patterns at at least 2 sample-detector distances has been developed. The method is very fast and is applicable for diffraction patterns without severe spot overlap, i.e. for sample with relatively few diffracting grains of identical or almost identical crystallographic orientations within the gauge volume. For further information see [eg.1, 2].

The spatial resolution of the 3DXRD is at present 1 μm x 5 μm x 5 μm for complete mapping of microstructures. However, microstructural elements down to 100 nm can be recorded provided they have an appropriate orientation difference to the rest of the microstructure, but it is not possible to map the position of such an element to a precision better than the 1 μm x 5 μm x 5 μm.

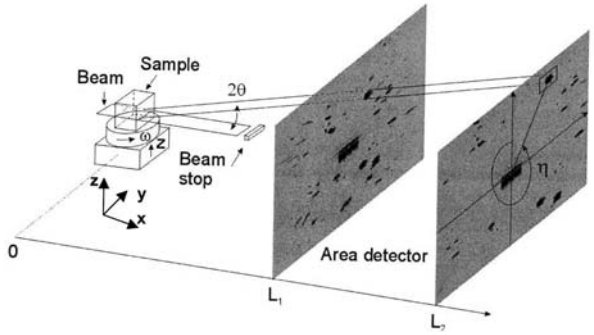

Fig. 1. Sketch of typical 3DXRD configurations. Diffraction patterns (examples shown in grey) are acquired on a high-resolution 2D detector positioned at several distances to the sample.

3. Example of applications

The 3DXRD microscope is now a standard ESRF instrument available to external users upon the usual peer review procedure. It has thus been used for a wide variety of investigations, including ceramic, geological and metallic materials as well as ice. Her only 3 examples will be resumed.

Plastic Deformation. Upon plastic deformation the individual grains in a polycrystalline material has to deform and their crystal lattice rotate in order to accommodate the macroscopic deformation. It is well known how the grain ensemble on average rotates (i.e. how the texture changes) but it is not known, how the individual grains in the bulk sample rotates, and what the influences of the neighbouring

grains are. This information is important for texture and thus property improvements in a range of products.

3DXRD can provide real-time observations of grain rotations in bulk materials during the deformation process. Experimentally, this is done by following the rotation of the diffraction spots as a function of strain [3, 4]. Conclusions from this work are that none of the existing texture models correctly predict the rotation of the all individual grains and that the influence of the orientation of a grain itself is significantly more important than that of its neighbours [4]. The latter conclusion is remarkable as a major trend in today's texture modelling is to extend models to include effects of neighbouring grains.

Recrystallization. 3DXRD allows in-situ investigations of the recrystallization kinetics of individual embedded grains [5]. More spectacularly it also allows direct "filming" of the growth of a single grain in the bulk of a deformed sample [6]. A few snapshots from a film are shown in Fig. 2. Besides giving full information about the local migration rates at every point of the boundary, the data reveals that the growth of the grains typically are very inhomogeneous, the shapes of the evolving grains can be highly irregular and the boundary movement may be jerky [6]. This contradicts theoretical predictions, which for the investigated example are steady-state motion and spherical grain shape.

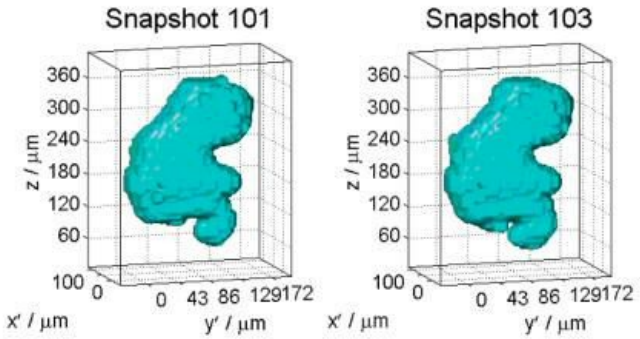

Fig. 2. growth of a nuclei within a 20% deformed aluminium single crystal of {110} <001> orientation.

Phase Transformations. 3DXRDallows in-situ studies of the nucleation and growth during phase transformations. Results of this type are published in [7]. Also in this case significant deviations from classical models were observed, which were intrepreted in terms of differences in the local environment including gradients in the local chemestry [7].

4. Outlook

3DXRD is a new technique and its unprecedented potentials for materials science investigation are definitely not fully explored. Within the topics, where it has been used to map the local bulk microstructural development, it is generally found that existing models do not describe correctly to local phenomena. This calls for many more experiments on well-planned models systems as well as more statistics on specific phenomena in order to improve existing theories or to develop new ones. Following the experimental needs the instrumental specifications are expected to change. Presently, work is focussed on developing

- A fully integrated data acquisition and analysis system.
- An improved area detector with a spatial resolution of 2x2x1 μm^3.
- The beamline at which the 3DXRD microscope is situated is undergoing a complete refurbishment, with the aim of pushing the methodology toward the nano-regime. Hence, by 2006 a so-called 3DXRDnanoscope is to be commissioned.

5. Acknowledgements

The authors gratefully acknowledge the Danish National Research Foundation for supporting the Center for Fundamental Research: Metal Structures in Four Dimensions, within which this work was performed, as well as the Danish National Science Research Council, who supported the work via DANSYNC. A special thanks to Ms. Eva Nielsen for her assistance with the manuscript.

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